SULFITE PULPING EXPERIMENTS ON SAND PINE

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Summary

Several sand pine (Pinus clausa) bolts with an average density of 32 pounds per cubic foot, average growth rate of 9.6 rings per inch, and average heartwood volume of 38.8 percent, were separated into heartwood and sapwood for sulfite pulping experiments. The sapwood had chemical characteristics similar to those of the whole wood of sand pine samples previously received at the Forest Products Laboratory, whereas the heartwood was found to be low in lignin and cellulose contents and exceptionally high in materials soluble in alcohol-benzene, ether, 1 percent sodium hydroxide, and hot water.

Using moderate pulping conditions, the sapwood chips were completely digested to a low bleaching pulp, but the heartwood chips were incompletely reduced, two-thirds of the total fibrous yield of 57 percent became screening rejects. Application of a relatively low maximum temperature and a long digestion time improved the heartwood pulping somewhat, but even under these favorable conditions the screening rejects amounted to two-fifths the total yield.

The sapwood pulp of sand pine had a chlorine demand of only 2.9 percent, had much lower strength properties than sapwood pulps obtained from other southern pines and was high in cellulose and rather high in ether extractive contents. The heartwood pulps had high chlorine demands, had inferior strength properties, were high in lignin and low in cellulose, and were exceptionally high in extractives. The chemical constituents of the sapwood and heartwood pulps were consistent with those of the chips before digestion.

Introduction

Sand pine is a minor member of the southern yellow pine growing in Florida and Alabama. A stand of sand pine of some 400,000 cords, capable of supporting an annual cut of 40,000 cords on a 35-year rotation, grows in the Ocala National Forest in north central Florida. It has little lumber value, but finds some outlet as sulfate pulp. Wells and

Rue[1] reported that sand pine could be readily reduced by the sulfite process to bleachable pulps in the yield range of 25 to 35 percent. This appears to be the only reference available on the sulfite pulping of this species. The possibility of making a wide variety of kraft papers including wrapping, high-grade paper, and newsprint was recently reported.[2]

The limited investigation reported here was undertaken at the request of the Ocala National Forest to determine the feasibility of pulping sand pine by the sulfite process using commercial conditions. The experiments were conducted on separated heartwood and sapwood samples and included: (1) digestions of both the heartwood and sapwood under moderately mild pulping conditions and (2) a digestion of the heartwood using a rather low maximum temperature and a long digestion time.

Experimental Part

Six 4-foot bolts of sand pine arrived at the Forest Products Laboratory from the Ocala National Forest on June 24, 1942, and were designated as shipment No. 1667. One badly decayed bolt was discarded. Most of the outer bark of all bolts had been knocked off during shipment. The inner bark had been penetrated with borers and beetles and serious blue stain extended to the heartwood. One-inch discs were cut from the center of each bolt for the physical tests. After the bark had been completely removed the wood was separated into heartwood and sapwood portions and converted into 5/8-inch chips. The two kinds of chips were sampled for chemical analyses.

The digestions were made in a stainless-steel autoclave with a steam jacket. Its capacity was 1.5 cubic feet. The pulped chips were defibered in a stirrer and passed through a diaphragm screen equipped with plates having 0.012 inch slots. The digester charge was 15.3 pounds for the sapwood and 16.8 pounds for the heartwood, both on a moisture-free basis. The acid charge was 7.0 gallons. Other digestion conditions are discussed later in this paper.

Results and Discussion

The Wood

The bolts ranged in diameter from 7 to 9 inches. The physical characteristics of this shipment were similar to those of a previous shipment, No. 1375, except for a somewhat higher density, as shown in table 1. The higher density figure was within the range of densities for most southern pines.

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[1] Wells, S. D. and Rue, J. D. U.S.D.A. Bulletin 1485 (1927).

[2] Bray, M. W. and Martin, J. S. South. Pulp & Paper Jour., June 1942.

The chemical composition of the sapwood from shipment No. 1667, given in table 2, showed good agreement with that of the whole wood from shipment No. 1375, although shipment No. 1375 appeared to be unusually low in pentosans and ether extractive material. The heartwood from No. 1667 differed greatly from its own sapwood or the whole wood of the previous shipment. It was low in lignin and alpha and total cellulose contents and high in material extracted by alcohol-benzene, ether, 1 percent sodium hydroxide, and hot water. The high caustic solubility value indicated the presence of decay but none was detected visually.

Pulping Experiments

Because the physical and chemical characteristics of previous shipments of sand pine were similar to those of northern jack pine (Pinus banksianna), pulping conditions known to be fairly satisfactory for jack pine were used for the first experiments. The application of these moderately mild conditions to the sand pine sapwood resulted in complete pulping and a very low bleaching pulp. This is shown by digestion No. 381-Y in table 3. The screening rejects of approximately 1 percent were probablly due to the presence of a small proportion of heartwood. The sapwood pulp, which was slightly overcooked, had very poor strength properties. It was much weaker than sulfite pulps from other southern pines. It was high in both total cellulose and alpha cellulose content. A higher content of ether soluble material than desirable for satisfactory paper making was present. It does not appear from these results that the sapwood, even if it can be readily obtained free from heartwood, is a promising material for sulfite pulping under standard commercial conditions. This does not preclude the possibility that more suitable pulping conditions can be developed to improve the qualities of the sapwood pulp.

The heartwood, when digested under the conditions used for the sapwood, was not successfully pulped, as shown by the data for No. 380-Y in table 3. The bisulfite in the cooking liquor was exhausted about 2 hours sooner than in the sapwood digestion, indicating the presence of the bisulfite-destroying components that are typical of pine heartwood. The exhaustion of the bisulfite and consequently the shortened pulping time caused incomplete pulping and gave evidence of an extremely refractory material. Approximately two-thirds of the total fibrous yield was in the form of screenings. The screened heartwood pulp was high in lignin and extractives and low in total and alpha cellulose (table 4). It, therefore, had a high bleach requirement as shown by the chlorine requirement in table 3. It was very weak from a paper-making standpoint (table 4).

Since a low maximum temperature is known to be favorable in the pulping of refractory heartwoods, a second digestion was made of the heartwood in which the maximum temperature was decreased from 136° to 130° C. This change lowered the rate of the reaction and resulted in an increase in digestion time from 11.3 to 14.5 hours and a small improvement in the degree of pulping. Although the screening rejects were half as much as in the first heartwood digestion,

they still amounted to two-fifths of the total yield. The chlorine requirement of the pulp was slightly lower than for the first heartwood digestion. Slight improvements in strength properties and chemical purity were realized. The pulp, however, was still inferior in quality.

Conclusions

The sapwood from a small sample of sand pine was completely pulped by the sulfite process under conditions comparable to those of commercial practice; under similar conditions the heartwood was only partially pulped. The pulps were decidedly inferior in paper-making qualities compared to sulfite pulps from other southern pines. Therefore, sand pine is not indicated to be a promising source of sulfite pulp as ordinarily prepared.

Table 1.--Physical characteristics of sand pine

	Shipment number	
	1667[1]	1375
Diameter (inches)	7.94	5.9
Age (years)	38.2	34.7
Growth rate (rings per inch)	9.6	12.2
Volume of:		
Heartwood (percent)	38.8	37.1
Summerwood (percent)	32.5	....
Density[2] (pounds per cubic foot)	32.0	28.6

[1]Average for 5 bolts, none containing decay.

[2]Oven-dry weight and green volume.

Table 2.--Chemical analyses of the sapwood and heartwood of sand pine

	Shipment 1667		Shipment 1375
	Sapwood	Heartwood	Whole wood
	Percent		
Lignin	26.8	24.0	27.2
Cellulose:			
Total	60.5	48.0	61.0
Alpha	46.6	35.6	42.9
Pentosans:			
Total	11.4	11.8	6.9
In cellulose	10.8	11.8	....
Solubility in:			
Alcohol benzene	3.0	17.7	2.2
Ether	1.9	14.0	1.1
1 percent sodium hydroxide	9.2	25.0	11.8
Hot water	2.4	7.0	2.5
Ash	.3	.2	....

Table 3.--Sulfite pulping of sand pine sapwood and heartwood

Digestion	Wood		Cooking acid		Penetration		Cooking				Total time	Yield		Chlorine requirement
	Kind	Dryness	Total SO_2	Comb. SO_2	Time to 110° C.	Time at 110° C.	Max. temp.	Time to max. temp.	Time at max. temp.			Total	Screenings	
Number		Percent	Per-cent	Per-cent	Hours	Hours	°C.	Hours	Hours		Hours	Per-cent	Percent	Percent
381-Y	Sap-wood	69.4	6.76	1.35	1.5	2.0	136	8	5.2		13.2	46.2	0.9	2.9
380-Y	Heart-wood	76.1	6.37	1.35	1.5	2.0	136	8	3.3		11.3	56.9	34.4	16.3
382-Y	Heart-wood	76.5	7.10	1.36	2.0	2.0	130	10	4.5		14.5	53.0	19.8	12.6

Table 4.--Physical properties and chemical analyses of sand pine sapwood and heartwood sulfite pulps

Digestion number and material	Physical properties of beaten pulp test sheets--25 by 40 - 500 ream										Chemical analyses							
	Bursting strength		Tearing strength		Tensile strength		Solid fraction		Beating time		Lignin	Cellulose		Total pentosans	Solubility in--			Ash
	Freeness-S.R.		Freeness-S.R.		Freeness-S.R.		Freeness-S.R.		Freeness-S.R.			Total	Alpha		Ether	Alcohol-benzene	NaOH	
	800 cc.	550 cc.	800 cc.	550 cc.	800 cc.	550 cc.	800 cc.	550 cc.	800 cc.	550 cc.								
	Pts. per lb. per ream		Gm. per lb. per ream		Lb. per sq. in.				Min.		Per-cent	Per-cent	Per-cent	Percent	Per-cent	Percent	Per-cent	Per-cent
381-Y Sapwood	0.36	0.39	0.75	0.50	3,600	4,600	0.46	0.55	17	40	0.8	96.0	80.4	4.6	2.2	2.5	8.4	0.4
380-Y Heartwood	.27	.32	.60	.40	2,100	3,700	.37	.45	8	27	9.0	74.2	61.1	4.0	15.2	16.6	25.7	0.9
382-Y Heartwood	.29	.37	.65	.45	2,200	4,100	.37	.46	15	44	8.1	75.5	64.0	4.0	15.5	15.8	22.7	0.8

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